Short communication

Mixed ion and electron conductive composites for single component fuel cells: I. Effects of composition and pellet thickness

Liangdong Fan [a,b,*], Chengyang Wang [b], Ose Osamudiamen [a], Rizwan Raza [a,c], Manish Singh [a], Bin Zhu [a,*]

[a] Department of Energy Technology, Royal Institute of Technology (KTH), S-10044 Stockholm, Sweden
[b] Key Laboratory for Green Chemical Technology of Ministry of Education, School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, PR China
[c] Department of Physics, Comsats Institute of Information Technology, Lahore 54000, Pakistan

HIGHLIGHTS

► SCFCs are assembled by mixed ion and electron conductive composites.
► The performances are influenced by the composition and thickness of the pellets.
► A maximum power density of 350 mW cm^{-2} is achieved at 550 °C.
► The performance is correlated with the mixed conduction (ionic and electronic, p and n-type) properties and the porous microstructure.

ARTICLE INFO

ABSTRACT

Electrochemical performances of single component fuel cells (SCFCs) based on mixed ion and electron conductors have been studied as a function of composition and pellet thickness by polarization curves and electrochemical impedance spectroscopy. The electronic conductor of LNCZO shows conductivities of 21.7 and 5.3 S cm^{-1} in H$_2$ and in air, respectively. SCFC using 40 wt. % of LNCZO and 60 wt. % of ion conductive SDC-Na$_2$CO$_3$ with a thickness of 1.10 mm shows the highest power density of 0.35 W cm^{-2} at 550 °C. The performance is correlated to the mixed conduction properties (ionic and electronic, p and n-type) and the microstructure of the functional SCFC layer.

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1. Introduction

Novel energy conversion and storage technologies have attracted more attention in recent years because of the increasing energy demands and severe environmental pollution. Fuel cells are promising devices for clean and efficient power generation. Among the diverse types of fuel cells, the proton exchange membrane fuel cells and solid oxide fuel cells (SOFCs) show the greatest potential when the versatile applications are taken into consideration. However, the commercialization of these green technologies is still far to be realized. Taking SOFC as an example, high temperature

operation needs chemically and thermally stable cell components, as well as their compatibility because of the complex structures, which cause the high cost and durability problems. Even though the updated materials and technologies have significantly reduced the gap, its cost and lifespan are still far from market demands.

Recently, a single component fuel cell (SCFC) or electrolyte free fuel cell was invented and developed [1–7], and has fascinated great attention [8,9]. The novel SCFC without the usage of the electrolyte and the classic three-component structure can still realize the fuel cell function only with one layer mixed ion and electron conductive materials. It had ever been reported earlier about the construction of SCFC using a single layer of the La$_{0.9}$Sr$_{0.1}$InO$_{3-\delta}$ by He et al. [10,11]. However, there still exists an electrolyte layer, in which the ion passing through is the key point to realize the energy conversion as same as the conventional SOFCs. This differs substantially from the SCFC reported by Zhu and the coworkers [1–6]. With only one homogeneous mixing conducting

* Corresponding authors. Department of Energy Technology, Royal Institute of Technology (KTH), S-10044 Stockholm, Sweden. Tel.: +46 8 7907403; fax: +46 8204161.
E-mail addresses: liafan@kth.se (L. Fan), binzhu@kth.se (B. Zhu).

layer based on the nanocomposite material, the SCFC in our case removes the mechanically and chemically compatible issues. It also provides technological advantages including simplified fabrication, the stack design and working requirement compared with the traditional SOFC's complex anode–electrolyte–cathode three-layer structure. All of these advantages will markedly lower processing costs and thus promote SCFC's practicability and commercialization.

The materials used in SCFC are a mixture of two kinds of composite materials: an ionic conductor and a semiconductor. The ionic conductors are the $Sm_{0.2}Ce_{0.8}O_{1.9}$ (SDC) or the doped ceria-salt (nano) composite materials [12–15]. While the semiconductors are normally transition metal oxides like NiO, CuO, ZnO and FeO_x, with or without lithiation. These transition metal oxides have been proven to show high catalytic activity for both hydrogen oxidation reaction and oxygen reduction reaction (redox reactions) [16–19]. Since the origination of SCFC in 2010, the basics and fundamental issues such as materials choice, structure and morphology, their redox reaction activities as well as the SCFC working principle (ionic conduction and charge separation), have been studied. However, all these undertakings are in the early SCFC research and development stage. Xia et al. [20] recently tried to optimize the electrical conductivity of the SDC-$Li_{0.15}Ni_{0.45}Zn_{0.4}O_x$ to balance the numbers of its ion and electron conductivities in SCFC by adjusting the relative weight percentage of the ionic conductor and semiconductor composite. As discovered that the pellets with 30 wt. % of $Li_{0.15}Ni_{0.45}Zn_{0.4}O_x$ displayed the highest conductivity of 0.1 S cm^{-1} at 600 °C based on the impedance analysis. However, their studies were carried out in air, which is a distinct environment from the fuel cell conditions, H_2/Air or H_2/O_2 atmosphere. In addition, the electrochemical performance of SCFC was not considered in their research.

In this work, we continue to optimize the electrochemical performances of SCFC by adjusting the composition of ionic and electronic conductor and pellets thickness or powder weight. The SCFC performances are characterized in H_2/air atmosphere by current density–voltage curves and current density–power density characteristics and complex electrochemical impedance spectroscopy. The results reported here are vital for the further research and development associated with SCFCs.

2. Experimental

The SDC-Na_2CO_3 nanocomposite electrolyte with enhanced ionic conductivity was used as the ionic conductor and prepared by co-precipitation technique as report elsewhere [17]. Semiconductor material of LiNiCuZnO was synthesized by solid-state reaction with Li_2CO_3, $NiCO_3 \cdot 2Ni(OH)_2$, $CuCO_3 \cdot Cu(OH)_2$ and $Zn(NO_3)_2 \cdot 6H_2O$ as the raw materials at the weight ratio of 1.5:2.5:7:7. The detailed preparation procedure could be referred to the literature [18].

To identify the effect of composition on the electrochemical performance of SCFC, composite powders with different mass percentages of semiconductor (major the electronic conductivity) were mixed with SDC-Na_2CO_3 first and sintered at 700 °C for 1 h. Then SCFC single pellet with a weight of 0.5 g was prepared by uniaxial dry-pressing under 200 MPa using a nickel foam as the mechanical support. To find the role of pellet thickness (varying of the sample weights), composite powder with a composition of 40 wt. % of electronic conductor and 60 wt. % of SDC-Na_2CO_3 was chosen. Powder weights of 0.3, 0.4, 0.5, 0.6 and 0.7 g were used respectively to vary the pellet thickness and prepare the pellets as the above described process. The thickness of the pellet using 0.5 g composite powder is around 1.10 mm. All the green pellets had a diameter of 13 mm. The surface of pellets without nickel foam was painted with silver paste (Shanghai Research Institute of Synthetic Resins, China) to collect current.

All the green pellets were in situ sintering during the temperature rising from ambient temperature to 600 °C and held for 30 min before being turned to the desirable temperatures for electrochemical measurements. Pure hydrogen gas was fed into "anode" side at a rate of 100 ml min^{-1} as fuel while the flowing air was used as the oxidant. Electrochemical performance measurements of SCFCs were conducted on the single cells after they reached the stable open circuit voltage (OCV). The voltage–current curves were recorded on a fuel cell tester (SM-102, Sanmu Co., China) by changing different external circuit resistance loading. The electrochemical impedance spectra were performed on an impedance analyzer VERASTA 4 (Princeton Applied Research, USA) under OCV condition with different compositions. The applied frequency was between 100 kHz and 0.1 Hz with an excitation amplitude of 20 mV. DC conductivities of LNCZO (pellet sintered at 700 °C for 1 h) in flowing H_2 and air, respectively, were measured between 600 °C and 300 °C with a ramping rate of 13.3 °C min^{-1}. They were measured by the four-probe method with the Digital Micro-ohm meter instrument (KD2531, China). The pellet was first fully oxidized or reduced in air/H_2, respectively, then the resistances (R) of the pellets were recorded. The conductivity (σ) was calculated by the following equation: $\sigma = L/(RS)$. Where L is the thickness of the targeted pellet (0.287 mm) and S is the active area of the pellet (0.78 cm^2). The microstructure images of pellets were taken by a scanning electron microscope (SEM, Zeiss, Germany).

3. Results and discussion

The electrical conductivities of LNCZO semiconductor both in pure hydrogen and in air are shown in Fig. 1. As reported before, NiO and CuO are well-known semiconductors with p type conductivity while the ZnO is an n-type conductor [4,5,21,22]. The electrical conductivities of these oxides are rather low. However, the lithiation reaction could notably improve their conductivities [21]. The electrical conductivities of LNCZO increase with the increase of temperatures both in air and in hydrogen. The $\ln(\sigma T)$ shows a linear relationship to 1000/T at the tested temperature range, indicating that LNCZO follows the semiconductor conductive behaviors [23]. Therefore, it can be inferred that LNCZO can preserve the major oxidation state even it could be reduced to some content in the reduced atmosphere. The value is 5.3 S cm^{-1} in air at 600 °C, which is close to the conductivity of lithiated NiO (around 10 S cm^{-1}) and ZnO doped lithiated NiO composite [18]. The conductivity reaches 21.7 S cm^{-1} at 600 °C in hydrogen gas, which is almost 3 times
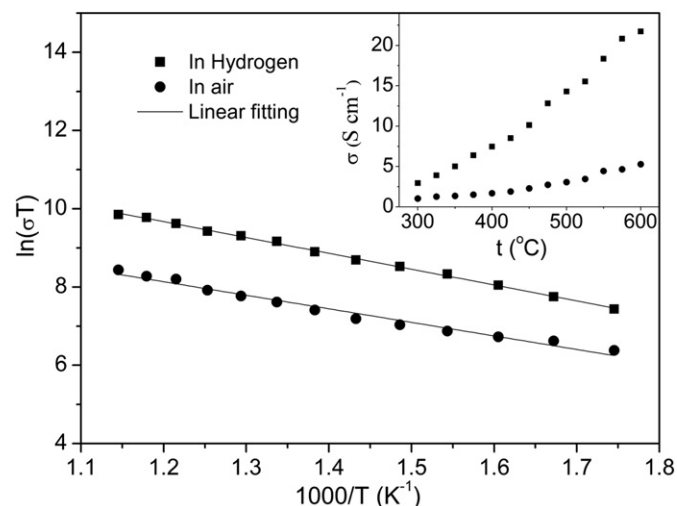

Fig. 1. Electrical conductivities of LNCZO in hydrogen and in air, respectively.

higher than that recorded in air. The results demonstrate that it is extremely necessary to investigate the separate conductivity of the electronic conductor in air and in a reduction atmosphere to find a balance point for mixed ion and electron conductive LNCZO/SDC-Na_2CO_3 composite [20].

The electrochemical performances of SCFCs at 550 °C are shown in Fig. 2. The pure SDC-Na_2CO_3 composite electrolyte pellet gives a OCV as high as 0.92 V, while the pure LNCZO gives a voltage of only 0.04 V, and both of them give negligible currents. In the former case, the SCFC was constructed by the SDC-Na_2CO_3 following the same protocol as given by He et al. [10,11]. The n-type conductivity can be caused by ceria-surface reduction at fuel side [24]; while at air side, p-type conductivity may be also formed [25]. However, due to negligible n and p conductivity formed and the relative thick electrolyte layer, this SDC-Na_2CO_3 based SCFC could not deliver observable current outputs. In the latter case, the electronic conductivity is much higher than the ionic one, serious mismatching between them, as demonstrated in Fig. 1, producing the nearly zero OCV. There is another consideration regarding of pure SDC-Na_2CO_3 pellet, the high OCV could be caused by the catalytic function of nickel foam and silver paste used as the current collects on the both surfaces. But the negligible current suggests the catalytic role of nickel foam and silver can be ignored because of the limited reaction active sites − triple boundary phases (TPBs). Actually, the catalytic function of nickel foam and silver paste has been a controversial topic since the invention of this new energy conversion device. In our previous experiments, performance of single cells without nickel foam and Ag paste (only the mixed conductive LNCZO and SDC-Na_2CO_3 composite layer) showed slightly lower performances for the inadequate conductivity (around 0.1 S cm^{-1}) of this mixed ion and electron conductive composite [2]. Simultaneously, these samples showed poor mechanical strength. Therefore, the negligible role of catalytic activity of nickel foam and Ag paste is again identified in this work.

Because of the low sintering temperature, the mechanical strength of pellet is inadequate to undergo the thermal expansion and possible redox cycling, while the employment of nickel foam can overcome the drawback and simultaneously can act as the excellent current collector in the fuel side. The influences of LNCZO content in composite powders on the OCV and power density of the SCFC are displayed in Fig. 3. As expected, the OCVs of pellets decrease with the increase of LNCZO contents. The OCV is 0.92 V for 30 wt. % LNCZO pellet, similar to pure nanocomposite electrolyte pellet. This can be interpreted by the percolation theory [26,27];
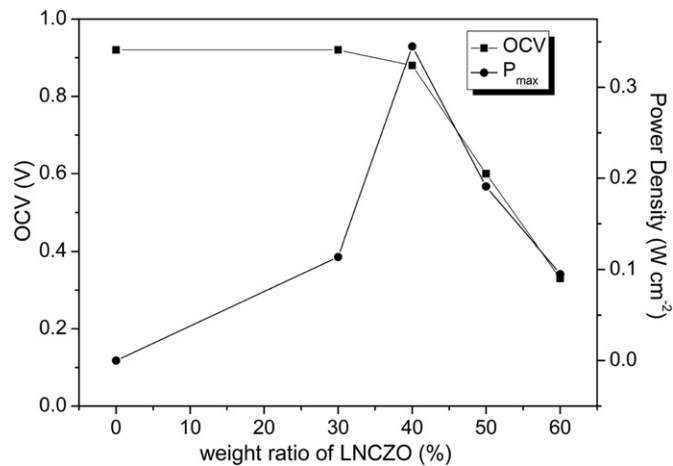

Fig. 3. Effects of LNCZO content on electrochemical performances (P_{max} and OCV) of SCFCs.

the volume of LNCZO does not reach the percolation framework. While it gradually decreases to 0.87 V with 40 wt. % LNCZO and visibly reduce to 0.61 V with more LNCZ contents. The maximum power density of the pellets increases first up to 0.35 W cm^{-2} with a 40 wt. % of the LNCZO; then declines with the further addition of the LNCZO. Therefore, this is a tradeoff between OCV and maximum power density. Besides, all the I−V curves show linear relationship, the absolute value of the slope of the line is the total area specific resistance (ASR) value of the pellet. It therefore can be clearly found that the ASR reduces with the increase of LNCZO content because of the much improved electrical conductivity.

The electrochemical impedance spectra (EIS) of the SCFC with 0, 30%, 40% and 100% of LNCZO under H_2/air atmosphere are recorded under OCV condition and shown in Fig. 4 and Fig. 5. Firstly, we check the electrochemical response of the pure SDC-Na_2CO_3 composite electrolyte (0 wt. % LNCZO) in H_2/air atmosphere (real fuel cell condition). As can be seen from Fig. 4, the EIS contains two typical depressed arcs. The high frequency arc is associated with the bulk resistance of the electrolyte, while the low frequency arc is related to the grain boundary resistance. In general, for single phase electrolytes, the grain boundary resistance is 2−7 orders of magnitude higher than bulk (grain) resistance with respect to the structural factors: the segregation of dopants and the reduction of
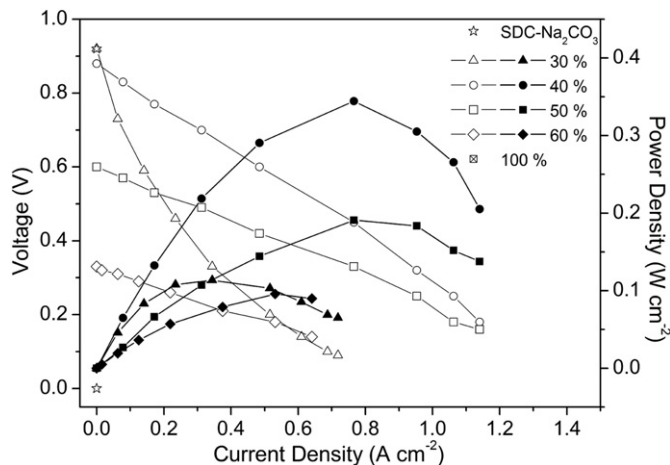

Fig. 2. LNCZO weight percentage dependence of I−V and I−P characteristics of SCFCs at 550 °C.
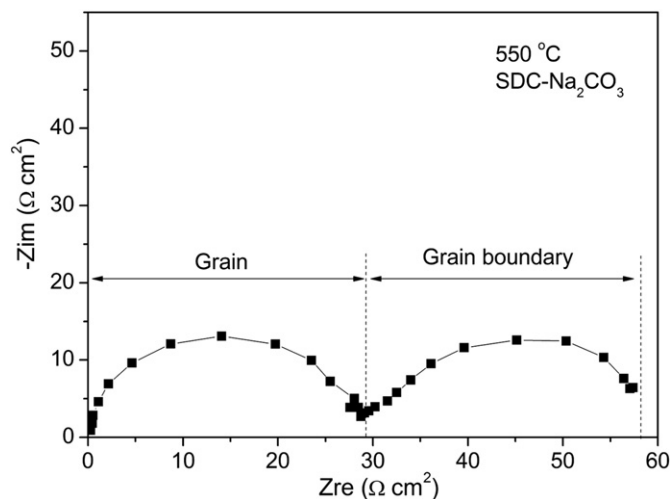

Fig. 4. Typical electrochemical impedance spectroscopy of ionic conductor of SDC-Na_2CO_3 under H_2/air atmosphere at 550 °C.
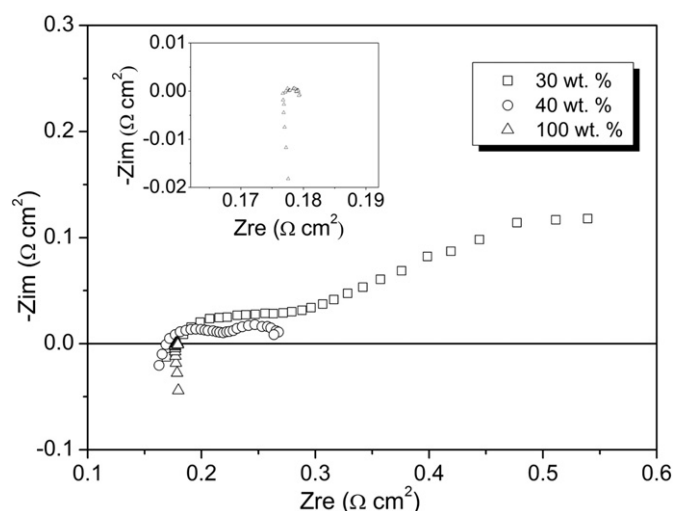
Fig. 5. Electrochemical impedance spectra of SCFCs with different LNCZO contents under open circuit voltage conditions at 550 °C.

oxygen vacancies along grain boundary [28,29]. Therefore the grain boundary resistance dominates the total impedance. While in this case, the grain boundary resistance shows close value to the bulk resistance for ionic transport in the SDC-Na$_2$CO$_3$ composite electrolyte. This could be probably explained by two aspects. One is the composite effect and/or the interfacial highway conduction mechanism which is always used to explain the enhanced conductivity on the composite electrolyte [30–33]. The nano nature of composite particles can provide a large amount of the surfaces and interfaces which are homogeneously distributed for quickly ionic transportation. The other one is the extrinsic nature of SDC-carbonate composite electrolyte − proton conduction in H$_2$ or H$_2$O contained atmospheres [34–36]. It has been reported that significant percentage of proton transported in SDC-carbonate composite electrolyte effectively enhances the total ionic conductivity, and hence the improved performance [34,36]. However, taking the non-bulk proton conductivity in SDC and carbonate at the high temperature (550 °C) into account, the interfacial conduction is the only probably explanation for the enhanced grain boundary conductivity. The study hence presents additional evidence for the super-ionic conduction in ceria-based composite electrolyte, especially the significant reduction of the grain boundary resistance.

The complex impedance spectra of pellets with different LNCZO weight content (30, 40 and 100 wt. %) are displayed in Fig. 5. The intercept of high frequency on the real axis is assigned to ohmic resistance, while the low frequency intercept reveals the total polarization resistance of fuel cell. Therefore, the difference between high frequency and low frequency intercepts is the electrode polarization losses, which is closely associated with the electrode reactions [37,38]. As can be seen from the inset of Fig. 5, a quite small arc is shown in the EIS of pure LNCZO, indicating the high catalytic activity for the redox reactions. The spectra of pellets with mixed conductive LNCZO-SDC composite contain two well identified depressed arcs while pure LNCZO pellet spectroscopy only has one small arc. Therefore, it can be concluded that the electrode reaction mechanism is changed when the ion conductive SDC-Na$_2$CO$_3$ is introduced. While the single component was situated against H$_2$ and air on its respective surface, the anode (H$_2$ contact side) and the cathode (air contact side) functions were formed on the component surfaces in situ FC condition. The addition of SDC-Na$_2$CO$_3$ could effectively enlarge the TPBs for electrode reactions and improve the ionic conduction. The electrode

polarization resistance decreases with the increase of the content of semiconductor LNCZO. We can see from Fig. 5 that there is a big polarization resistance difference between a pellet with 30 wt. % LNCZO and the pellet using 40 wt. % LNCZO. A continuous channel for electronic transport cannot formed because the electronic conductor in composite cannot reach the percolation threshold (~30 vol. %). However, with the increase of weight ratio of the electronic conductor to 40 vol. %, the electron produced in the electrode can be effectively transferred to the external circuit. This results in a much lower electrode polarization resistance, from several Ω cm^2 to 0.16 Ω cm^2, and thus a much better power density.

Fig. 6 displays the electrochemical performances of the SCFC pellets with various thicknesses (0.70, 0.88, 1.10 and 1.45 mm, respectively) by adjusting the total weights of composite powders (0.3, 0.4, 0.5 and 0.7 g, respectively). The OCV rises when the total powder weight increases, from 0.68 V to 0.91 V. While the maximum power densities (P_{max}) do not follow this trend. The SCFCs show close P_{max} values (around 0.2 W cm^{-2}) when the total weight of powder is less than 0.4 g. Then the P_{max} is improved remarkably to 0.35 W cm^{-2} with a cell thickness of 1.10 mm. However, the power output reduces to 0.17 W cm^{-2} when a further increase of the pellet thickness to 1.45 mm. All the tested pellets are in situ sintered at 600 °C, which is much lower than the sintering temperature, so large amounts of residual porosities are remained. The porous structure is on one hand required for completing FC reactions and high power outputs; on the other hand, it may cause a gas crossover to some extent between fuel and oxidant to decline OCVs and power outputs. Therefore, optimization of the porous structure is one of the key parameters to improve the SCFC performance, which deserves more continuous efforts in this aspect. Our initial efforts on this aspect in optimization by varying the thickness (weight) have reached a good balance point. The SCFC with 0.5 g composite powder showed the maximum power density, 0.35 W cm^{-2} at an OCV of 0.89 V. Though the SCFC with 0.7 g powder showed even higher OCV, 0.91 V, but its power output only reaches 0.17 W cm^{-2}. It should be noted that the performance in the present study is lower than the values reported before, but there still maintains large room for further optimization of various parameters. In previous efforts we had discovered that, by adding some higher active catalysts like the redox FeO$_x$ [2,4] and keeping the balance of ionic and electronic conduction, the fuel cell performance could be significantly improved. In addition, suitable cell fabrication technologies like *Spark Plasma Sintering* (*SPS*) technology for optimizing the microstructure/porous structure as
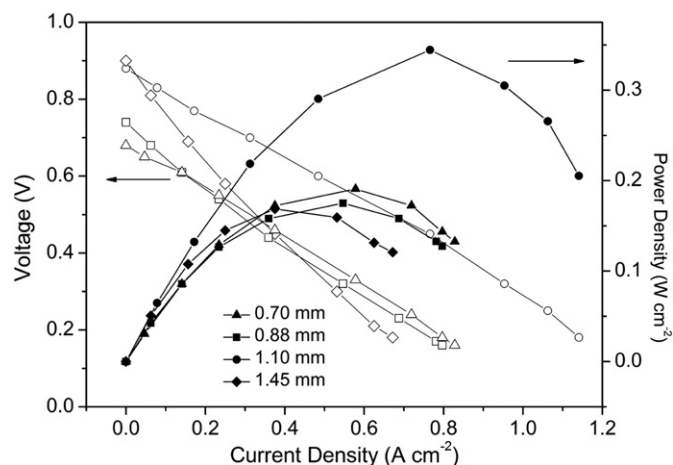

Fig. 6. Electrochemical performances of SCFCs with various pellet thicknesses at 550 °C. The weight ratio of LNCZO to SDC-Na$_2$CO$_3$ is fixed at 2:3.
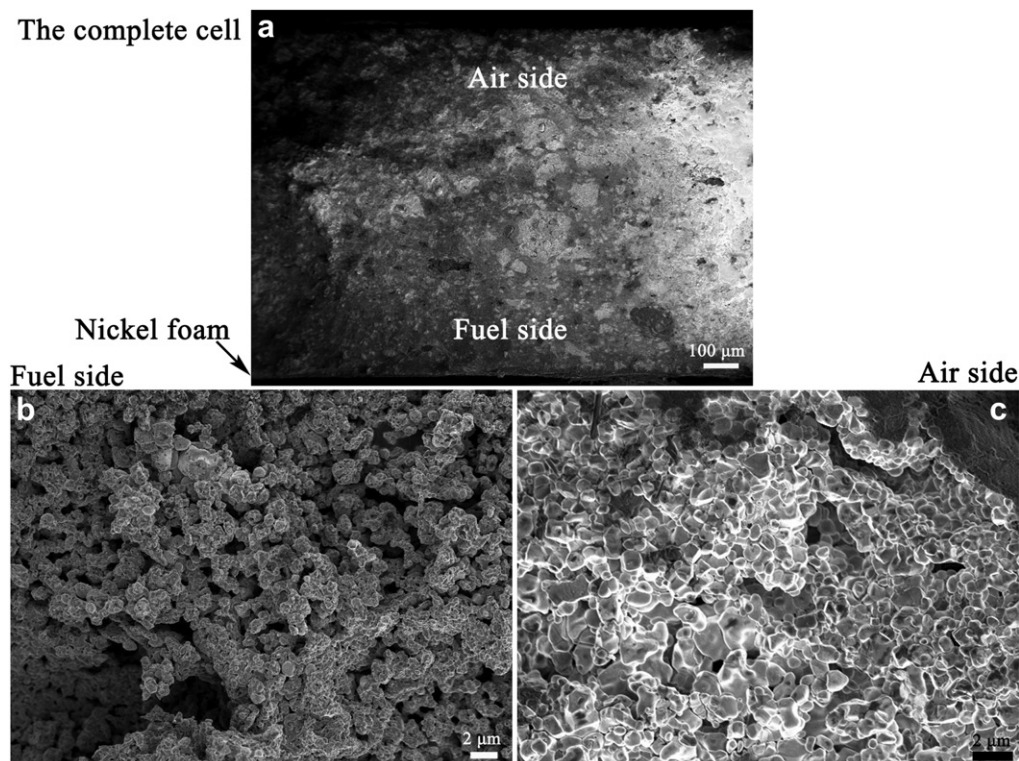
Fig. 7. SEM images of the fractural cross-section of the tested SCFCs: (a) the complete cell, (b) fuel side and (c) air side.

well as improving materials preparation procedure can be also adopted.

Fig. 7 reveals the microstructure features of SCFC after performance measurements. LNCZO and SDC-Na$_2$CO$_3$ are homogeneously distributed in complete cell as shown in Fig. 7a. Compared with the conventional SOFC, SCFC shows a much simpler fractural microstructure, only one functional layer can be seen except the current collector Ag paste and the nickel foam support. The magnified fuel side (Fig. 7b) and air side (Fig. 7c) show different morphologies after fuel cell operation. The surface exposed to the air appears smoother than that exposed to the fuel, which is probably caused by the partial reduction of fuel side particles leading to more porosity on the "anode" side. The porous structures of both cell sides will facilitate the mass transfer of the reactants and products in the electrode to deliver high power outputs. Besides, there appears to be more charging accumulates on the air side compared with the fuel side, suggesting that the conductivity of LNCZO mixed with ion conductor SDC-carbonate at oxidation state is lower than that at reduced state. It also indicates that the electrical conductivity of composite powder is inadequate in air. Therefore, a suitable current collector should be adopted to achieve a good performance.

4. Conclusions

Further performance optimizations of SCFCs were conducted in this present work. The effects of the weight ratio of ion and electron conductors and the pellets thickness on the electrochemical performances of SCFCs were systematically studied. A highest power density of 0.35 W cm^{-2} was achieved for SCFC using 40 wt. % of LNCZO − 60 wt. % of SDC-carbonate composite powder with a thickness of 1.10 mm. This study contributes to better understanding of the significant roles of the percolating electron and ion conductive phases, the balance of p and n-type conductions and the

microstructure of pellets on the electrochemical performance of SCFCs.

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